SOME STUDIES ON ALUMINIUM MATRIX IN-SITU COMPOSITES PRODUCED BY STIR CASTING METHOD

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF

Bachelor of Technology

In

Metallurgical & Materials Engineering

By

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CERTIFICATE

This is to certify that the thesis entitled,"SOME STUDIES ON ALUMINIUM MATRIX IN-SITU COMPOSITES PRODUCED BY STIR CASTING METHOD" submitted by Nigamananda Ray (10604036) and Dilip kumar kerketta (10604020) in partial fulfillment of the requirements for the award of Bachelor of Technology Degree in Metallurgical & Materials Engineering at the National Institute Of Technology, Rourkela is an authentic work carried out by them under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

Date: Dr.S.Sarkar

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CONTENTS

ABSTRACT

During the last two decades, metal matrix composites (MMCs) have emerged as an important class of materials for structural, wear, thermal, transportation and electrical applications. Metal matrix composites (MMCs) possess significantly improved properties including high specific strength; specific modulus, damping capacity and good wear resistance compared to unreinforced alloys. There are several techniques to produce composites, such as liquid state, solid state and semi solid state production route. Among the entire liquid state production route stir casting route are simplest and cheapest. The aim of our project is to produce a composite with cheap and simple production route i.e. stir casting method. We have chosen low grade iron ore as the reinforcement material and commercially pure aluminium as matrix phase. For the fabrication of in-situ composite wettability of the iron ore particles by liquid Al is essential. To improve wettability, Mg is added into Al melt .After fabrication; composites have been characterized for their morphology, mechanical and wear properties to see their suitability as a wear resistance material. Wear test was performed as a function of sliding distance, applied load, sliding velocity with the help of Pin-On-Disc wear test machine. The worn surfaces were analyzed using scanning electron microscope. The mechanical properties such as hardness have been investigated.

Key words: damping capacity, in situ chemical reaction and wettability, EDX analysis.

CHAPTER-I

1.0 INTRODUCTION:

A COMPOSITE MATERIAL is a macroscopic combination of two or more distinct materials, having a recognizable interface between them. [1] Composite is a multiphase material that exhibits a significant proportion of the properties of both constituent phases such that a better combination of properties is realized. This is termed as the principle of combined action. [2]

1.1 WHY USE COMPOSITES?

The composites industry has begun to recognize that the commercial applications of composites promise to offer much larger business opportunities than the aerospace sector due to the sheer size of transportation industry. Thus the shift of composite applications from aircraft to other commercial uses has become prominent in recent years.

The various reasons for the use of composites are due to

- To increase stiffness, strength and dimensional stability.
- To increase tough and impact strength.
- To increase heat deflection temperature.
- To increase mechanical damping.
- To reduce permeability to gases and liquids.
- To modify electrical properties.
- To reduce cost.
- To decrease thermal expansion.
- To increase chemical wear and corrosion resistance.
- To reduce weight.
- To maintain strength/stiffness at high temperatures while under strain conditions in a corrosive environment.
- To increase secondary uses and recyclability, and to reduce negative impact on the environment. [3]

1.2 CLASSIFICATION OF COMPOSITES:

Composites are commonly classified at two distinct levels. The first level of classification is usually made with respect to the matrix constituent. The major composite classes include organic-matrix composites (OMCs), metal-matrix composites (MMCs), and ceramic-matrix composites (CMCs). The term “organic-matrix composite” is generally assumed to include two classes of composites: polymer-matrix composites (PMCs) and carbon-matrix composites (commonly referred to as carbon-carbon composites). In each of these systems, the matrix is typically a continuous phase throughout the component. [1]

The second level of classification refers to the reinforcement form—particle reinforced, fiber-reinforced, and structural composites.

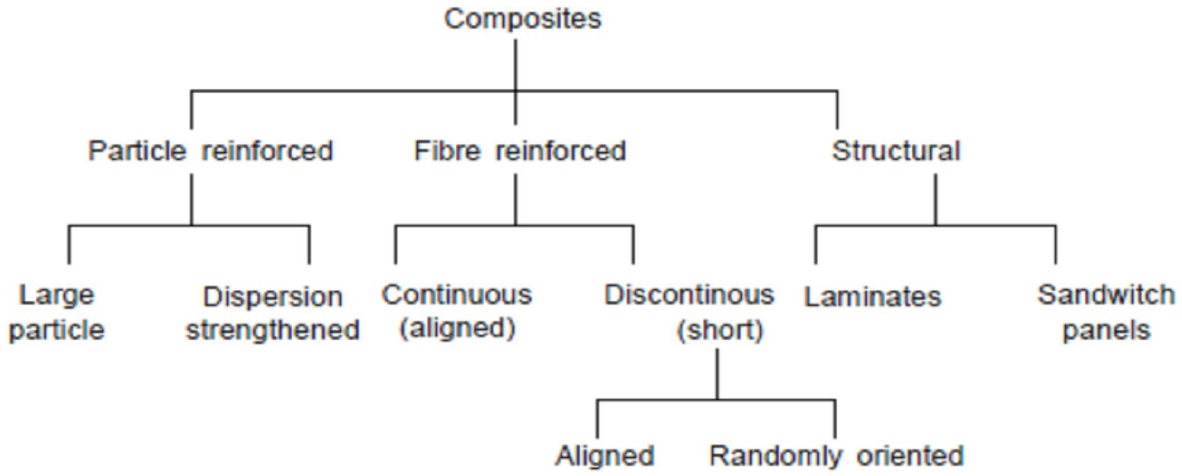

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Fig 1.1 A simple classification schemes for the various composite types[2]

The dispersed phase for particle-reinforced composites is equiaxed (i.e., particle dimensions are approximately the same in all directions) and the dispersed phase for fiber-reinforced composites has the geometry of a fiber-reinforced composites has the geometry of a fiber. (i.e. a large-length-to-diameter ratio). Structural composites are combinations of composites and homogeneous materials.

Examples of these three groups include concrete, a mixture of cement and aggregate, which is a particulate composite; fiber glass, a mixture of glass fibers imbedded in a resin matrix, which is a fiber composite; and plywood, alternating layers of laminate veneers, which is a laminate composite.[2]

1.2.1 PARTICLE REINFORCED COMPOSITE:

These can be further classified under two subgroups: (i) large particle and (ii) dispersion-strengthened composites. The distinction between these is based upon reinforcement or strengthening mechanism. [2]

1.2.1.1 LARGE-PARTICLE COMPOSITES:

The term large indicate that particle-matrix interactions cannot be treated on the atomic or molecular level. Properties are a combination of those of the components. The rule of mixtures predicts that an upper limit of the elastic modulus of the composite is given in terms of the elastic moduli of the matrix (E_m) and the particulate (E_p) phases by:

$$Ec = E_m V_m + E_p V_p$$

where V_m and V_p are the volume fraction of the two phases. A lower bound is given by:

$$Ec = E_m E_p / (E_p V_m + E_m V_p)$$

Concrete is a familiar example of large-particle composite. [2]

1.2.1.2 DISPERSION-STRENGTHENED COMPOSITES:

This type of composite contains small particulates or dispersions, which increase the strength of the composite by blocking the movement of dislocations. The dispersoid is typically a stable oxide of the original material. Particle-matrix interactions occur on the atomic or molecular level and lead to strengthening. Particles like oxides do not react so the strengthening action is retained at high temperatures. A common example is sintered aluminium powder (SAP).Particles for dispersion-strengthened composites are normally much smaller (diameter between 0.01 micrometer and 0.1 micrometer). [2]

1.2.2 FIBER-REINFORCES COMPOSITES:

These are strong fibers imbedded in a softer matrix to produce products with high strength-to-weight ratios. The matrix material transmits the load to fibers, which absorb the stress. The

length-to-diameter, or aspect, ratio of the fibers used as reinforcement influences the properties of the composite. The higher the aspect ratio, the stronger the composite. Therefore, long, continuous fibers are better than short ones for composite construction. However, continuous fibers are more difficult to produce and place in the matrix. Shorter fibers are easier to place in the matrix but offer poor reinforcement. There are two possible extremes with respect to orientation: (i) a parallel alignment of the longitudinal axis of the fibers in the single direction, and (ii) a totally random alignment. [2]

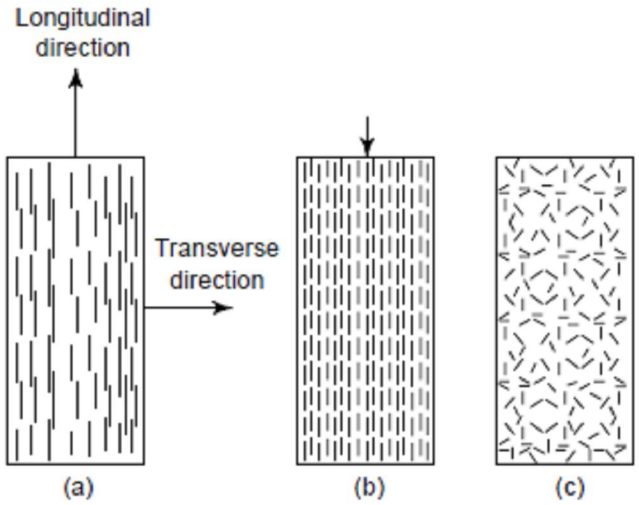


Fig 1.2 Representation of (a) Continuous and aligned fiber composites (b) Discontinuous and aligned fiber composites (c) Discontinuous and randomly oriented composites. [4]

1.2.3 STRUCTURAL COMPOSITES:

1.2.3.1 LAMINAR COMPOSITES

When multidirectional stresses are imposed within a single plane, aligned layers that are fastened together one on top of another at different orientations are frequently utilized. These are called laminar composites. These are generally designed to provide high strength and low cost at a lighter weight. A familiar laminar composite is plywood.

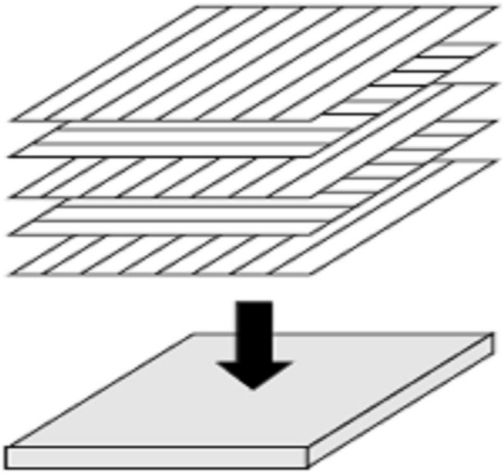


Fig 1.3 Laminar composite.[4]

1.2.3.2 SANDWITCH STRUCTURES

These have thin layers of facing materials over a low density material, or comb core, such as a polymer foam or expanded metal structure. A familiar sandwich-structured composite is corrugated cardboard. The corrugated paper core is covered by two faces of thin paper. In structures of this type, the facing material serves to fix the inner core in place. The core provides the strength. Typical face materials include aluminium alloys, fiber-reinforced plastics, titanium, steel and plywood. The popular core consists of a 'honeycomb' structure, which finds wide use in industries such as the aircraft industry, where higher strength and lower weight are important factors. The honeycomb structure consists of thin foils that have been formed into interlocking hexagonal cells, with axis oriented perpendicular to the face panels. [2]

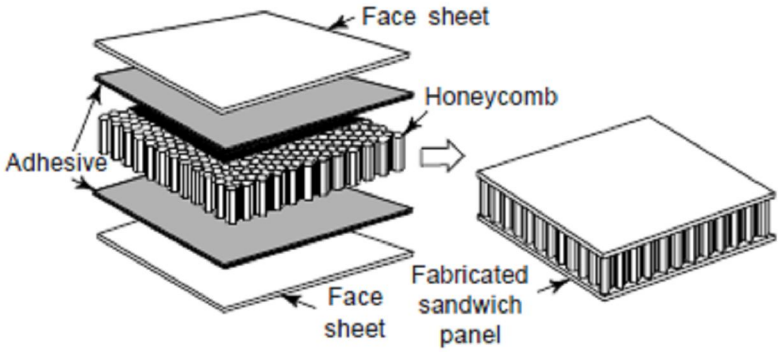


Fig 1.4 Honeycomb structure[4]

1.3 RULE OF MIXTURES

Rule of Mixtures is a method of approach to approximate estimation of composite material properties, based on an assumption that a composite property is the volume weighed average of the phases (matrix and dispersed phase) properties.

According to Rule of Mixtures properties of composite materials are estimated as follows:

Density

$$d_c = d_m * V_m + d_f * V_f$$

Where d_c, dm, df – densities of the composite, matrix and dispersed phase respectively;

V_m, V_f – volume fraction of the matrix and dispersed phase respectively.

Modulus of Elasticity

When two linear-elastic materials (though with different moduli) are mixed, the mixture is also linear-elastic. The modulus of a fibrous composite when loaded along the fiber direction is a linear combination of that of the fibers, Ef, and the matrix, Em.

$$E_{cl} = E_m * V_m + E_f * V_f$$

Where Vf is the volume fraction of fibers. The modulus of the same material, loaded across the fibers is much less – it is only

$$1/E_{ct} = V_m/E_m + V_f/E_f$$

Tensile Strength

- Tensile strength of long-fiber reinforced composite in longitudinal direction

$$\sigma_c = \sigma_m * V_m + \sigma_f * V_f$$

Where, σ_c, σ_m, σ_f – tensile strength of the composite, matrix and dispersed phase (fiber) respectively.

- Tensile strength of short-fiber composite in longitudinal direction

  (Fiber length is less than critical value L_c)

  $L_c = \sigma_f * d/\tau_c$

  Where

  d – Diameter of the fiber;

  τ_c –shear strength of the bond between the matrix and dispersed phase (fiber).

  $\sigma_c = \sigma_m * V_m + \sigma_f * V_f * (1 - L_c/2L)$

  Where

  L – Length of the fiber

- Tensile strength of short-fiber composite in longitudinal direction

  (Fiber length is greater than critical value L_c)

  $\sigma_c = \sigma_m * V_m + L * \tau_c * V_f/d$ [4]

1.4 METAL MATRIX COMPOSITES (MMCs)

The matrix in a metal matrix composite (MMC) is usually an alloy, rather than a pure metal, and there are three types of such composites, namely,

- dispersion-strengthened, in which the matrix contains a uniform dispersion of very fine particles with diameters in the range 10–100 nm,
- particle-reinforced, in which particles of sizes greater than 1 μm are present, and
- fiber-reinforced, where the fibers may be continuous throughout the length of the component, or less than a micrometer in length, and present at almost any volume fraction, from, say, 5 to 75%.[5]

Compared to monolithic metals, MMCs have:

- Higher strength-to-density ratios
- Higher stiffness-to-density ratios
- Better fatigue resistance
- Better elevated temperature properties
    - Higher strength
    - Lower creep rate
- Lower coefficients of thermal expansion
- Better wear resistance

The advantages of MMCs over polymer matrix composites are:

- Higher temperature capability
- Fire resistance
- Higher transverse stiffness and strength
- No moisture absorption
- Higher electrical and thermal conductivities
- Better radiation resistance
- No out gassing
- Fabric ability of whisker and particulate-reinforced MMCs with conventional metalworking equipment.

Some of the disadvantages of MMCs compared to monolithic metals and polymer matrix composites are:

- Higher cost of some material systems
- Relatively immature technology
- Complex fabrication methods for fiber-reinforced systems (except for casting)
- Limited service experience.

1.5 PROCESSING OF MMCs

Accordingly to the temperature of the metallic matrix during processing the fabrication of MMCs can be classified into three categories:

(a) Liquid phase processes,

(b) Solid state processes, and

(c) Two phase (solid-liquid) processes.

1.5.1 LIQUID METAL TECHNIQUES

In liquid phase processes, the ceramic particulates are incorporated into a molten metallic matrix using various proprietary techniques, followed by mixing and casting of the resulting MMC.

1.5.3.1 STIR CASTING METHOD OF FABRICATION OF MMCs

Stir Casting is a liquid state method of composite materials fabrication, in which a dispersed phase (ceramic particles, short fibers) is mixed with a molten matrix metal by means of mechanical stirring. The liquid composite material is then cast by conventional casting methods and may also be processed by conventional Metal forming technologies. [6]

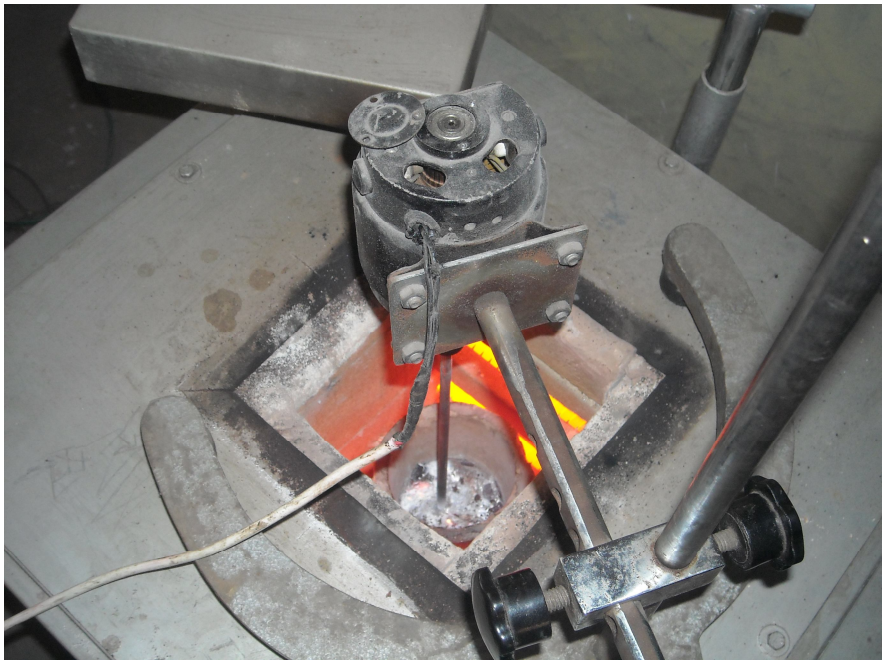


Fig 1.5 Laboratory stir casting set up

1.5.3.1.1 PROCESS VARIABLES AND THEIR EFFECTS ON PROPERTIES:

Speed of Rotation:

The control of mould speed is very important for successful production of casting. Rotational speed also influences the structure, the most common effect of increase in speed being to promote refinement and instability of the liquid mass at very low speed. It is logical to use the highest speed consistent with the avoidance of tearing.

Pouring Temperature:

Pouring temperature exerts a major role on the mode of solidification and needs to determine partly in relation to type of structure required. Low temperature is associated with maximum grain refinement and equiaxed structures while higher temperature promotes columnar growth in many alloys. However practical consideration limits the range .The pouring temperature must be sufficiently high to ensure satisfactory metal flow and freedom from cold laps whilst avoiding coarse structures.

Pouring speed:

This is governed primarily by the need to finish casting before the metal become sluggish; although too high a rate can cause excessive turbulence and rejection. In practice slow pouring offers number advantages. Directional solidification and feeding are promoted whilst the slow development of full centrifugal pressure on the other solidification skin reduces and risk of tearing. Excessive slow pouring rate and low pouring temperature would lead to form surface lap.

Mould Temperature:

The use of metal die produces marked refinement when compared with sand cast but mould temperature is only of secondary importance in relation to the structure formation. Its principal signification lies in the degree of expansion of the die with preheating .Expansion diminishes the risk of tearing in casting. In nonferrous castings, the mould temperature should neither be too low or too high. The mouid should be at least 25 mm thick with the thickness increasing with size and weight of casting.

Mould Coatings:

Various types of coating materials are used. The coating material is sprayed on the inside of the metal mould. The purpose of the coating is to reduce the heat transfer to the mould .Defects like shrinkage and cracking that are likely to occur in metal moulds can be eliminated, thus increasing the die life. The role of coating and solidification can be adjusted to the optimum value for a particular alloy by varying the thickness of coating layer. For aluminum alloys, the coating is a mixture of Silicate and graphite in water.

Mould Life:

Metal mould in casting is subjected to thermal stresses due to continuous operation. This may lead to failure of the mould. The magnitude of the stresses depends on the mould thickness and thickness of the coating layer, both of which influence the production rate. Deterioration takes place faster in cast iron mould than in steel mould. [7]

Stir Casting is characterized by the following features:

- Content of dispersed phase is limited (usually not more than 30 vol. %).
- Distribution of dispersed phase throughout the matrix is not perfectly homogeneous:

    1. There are local clouds (clusters) of the dispersed particles (fibers);

    2. There may be gravity segregation of the dispersed phase due to a difference in the densities of the dispersed and matrix phase.

- The technology is relatively simple and low cost.[6]

1.5.2 SOLID PHASE PROCESSES

Solid state fabrication of Metal Matrix Composites is the process, in which Metal Matrix Composites are formed as a result of bonding matrix metal and dispersed phase due to mutual diffusion occurring between them in solid states at elevated temperature and under pressure. [8]

1.6 STRENGTHENING MECHANISM OF COMPOSITES

The strengthening mechanisms of the composites are different with different kind of reinforcing agent morphology such as fibers, particulate or dispersed type of reinforcing elements.

1.6.1 DISPERSION STRENGTHENING MECHANISM OF STRENGTHENED COMPOSITE-

In the dispersion strengthened composite the second phase reinforcing agents are finely dispersed in the soft ductile matrix. The strong particles restrict the motion of dislocations and strengthen the matrix. Here the main reinforcing philosophy is by the strengthening of the matrix by the dislocation loop formation around the dispersed particles. Thus the further movement of dislocations around the particles is difficult. Degree of strengthening depend upon the several factors like volume % of dispersed phase, degree of dispersion, size and shape of the dispersed phase, inter particle spacing etc. In this kind of composite the load is mainly carried out by the matrix materials.

1.6.2 STRENGTHENING MECHANISM OF PARTICULATE COMPOSITE-

In the particulate reinforced composite the size of the particulate is more than 1 μm, so it strengthens the composite in two ways. First one is the particulate carry the load along with the matrix materials and another way is by formation of incoherent interface between the particles and the matrix. So a larger number of dislocations are generated at the interface, thus material gets strengthened. The degree of strengthening depends on the amount of particulate (volume fraction), distribution, size and shape of the particulate etc.

1.7 THE MECHANICAL PROPERTIES OF IN-SITU COMPOSITES:

In situ composites are multiphase materials where the reinforcing phase is synthesized within the matrix during composite fabrication. This contrasts with ex-situ composites where the reinforcing phase is synthesized separately and then inserted into the matrix during a secondary process such as infiltration or powder processing. In-situ processes can create a variety of

reinforcement morphologies ranging from discontinuous to continuous and the reinforcement may be either ductile or ceramic phases. The potential advantages of in-situ composites as compared to discontinuous metal ceramic composites produced by ex-situ methods include:

1. Smaller reinforcement particle size with higher strength (a contribution from composite strengthening mechanism) and improved fatigue resistance and creep.
2. Small, single crystal reinforcements (lower propensity for particle fracture).
3. Clean, un-oxidized particle matrix interfaces with higher interfacial strength (higher ductility and toughness) and improved wettability.
4. Thermodynamically stable particles that are weldable and castable do not dissolve at higher temperatures (vis-à-vis age-hardened alloys), and do not have a reaction layer (higher interfacial strength, improved corrosion and long term stability.)
5. Better particle size distribution (improved mechanical properties).
6. More conventional processing with the potential for lower cost and production with conventional equipment.

In conclusion, the objectives to develop the in-situ particulate composites can be summarized as follows:

1. The interfaces of the particle matrix can be cleaner, as the particles separate out of the matrix. It can lead to better interfacial bonding.
2. Very fine particles may form within the matrix to produce dispersion hardened particle composites.
3. The problem of non-wettability of particles and interface degradation by chemical reaction, as observed in synthetic composites can be ruled out.
4. The in-situ formed particles may be coherent with the matrix that may improve the mechanical properties of the composites.[9]

1.8 WEAR BEHAVIOUR

Perhaps the biggest in solving wear problems is that of anticipating the type(s) of wear to which components will be subjected. Material can be removed from a solid surface in only three ways: by melting, by chemical dissolution, or by the physical separation of atoms from the surface. The

last method can be accomplished either by the one-time application of a high strain or by cyclic straining at lower magnitudes. Mechanical and chemical processes may operate separately or together, such as abrasion in a corrosive medium. [12]

1.8.1 LAWS OF WEAR

The laws governing wear are not completely clear but both adhesive and abrasive wear a simple equation can be written as:

$V/X = K*(W/H)$

Where, V=Wear volume

X=Sliding distance

W=Normal load

H=Initial hardness of the softer sliding components

K= Wear coefficients

Further it has also been shown that there is relation between hardness H of the metal and its yield or flow pressure σy which given by: $H= 3* \sigma y$ From these equations we can conclude that the total volume of the metal removed due to sliding is directly proportional to applied loads, sliding distance and inversely proportional to hardness of the softer sliding component.

1.8.2 CALCULATION

Wear rate of the composite is related to mass loss (Δm) and sliding distance (L) as given below:

$Wr = \Delta m/L$

The volumetric wear rate Wv of the composite is related to density (ρ) and the abrading time (t) as given below:

$Wv = \Delta m / \rho t$

Specific wear rate expresses in terms of the volumetric wear rate as

Ws = Wv / Vs *Fn

Where Vs is sliding velocity

Fn is the applied load.

1.8.3 TYPES OF WEAR

Depending on the nature of movement of the media following types wear has been classified.

- Abrasive wear
- Sliding and Adhesive wear
- Corrosive wear
- Oxidational wear

1.8.3.1 ABRASIVE WEAR

ABRASIVE WEAR, as defined by ASTM, is due to hard particles or hard protuberances that are forced against and move along a solid surface. Wear, in turn, is defined as damage to a solid surface that generally involves progressive loss of material and is due to relative motion between that surface and a contacting substance or substances.

Effect of Environment on Abrasive Wear:-

In addition to the properties of a material, the environment affects wear. As stated earlier, abrasion loss rates are not intrinsic to a material. Environmental factors that effect abrasive loss include, but are not limited to: the type of abrasive and its characteristics, temperature, speed of contact, unit load of the abrasive on the material, humidity, and corrosive effects.

Abrasive:-

The hardness of the abrasive particles is important to the rate of abrasion of the subject material. As the hardness of the abrasive exceeds that of the wear material, abrasive wear typically becomes much worse .As the abrasive hardness exceeds the hardness of the material, it is able to

penetrate the surface and cut/remove material without having its cutting edges broken or rounded.

Temperature:-

It might be expected that abrasive wear would increase as the temperature rises, because the hardness and yield strength decrease. Instead, for aluminum and copper , when the temperature was increased from ambient to 673 K, very little change in the abrasive wear rate was observed. It has been proposed that the reason for this small change is that during abrasion, small areas are adiabatically heated. At higher initial temperatures, the metal flow stress is reduced. This results in less heating in the material during the abrasion process. The end result is that areas around the material that is being removed have a similar temperature, independent of starting temperature, and similar abrasion rates.

Speed of Contact:-

The rate of abrasive wear has been found to slightly increase with increasing speed in the range from 0 to 2.5 m/s (0 to 8.2 ft/s). This increase in wear may be attributable to frictional heating. The effect is small, because all of the abrasion occurs in a near-adiabatic process. This should result in nearly the same peak temperature rise, independent of speed, for the tiny volume of material where the asperities are removing the material.

Load:-

When load causes fracture of abrasive particle then wear can increase. If the abrasive particle points are rounded, wear will decrease.

Humidity:-

The effect of atmospheric humidity on abrasive wear is far from clear, and contrary results exist. Larsen- Basse studied the effect of atmospheric humidity on abrasive wear for a variety of pure metals and steels. When using SiC abrasive, wear usually increased with increasing humidity, up to 65% relative humidity. This increase is attributed to a moisture-assisted fracture of the SiC abrasive particle, which resulted in fresh sharp edges to cut into the surface of the material.[10]

1.8.3.2 SLIDING AND ADHESIVE WEAR

Both refer to a type of wear generated by the sliding of one solid surface along another surface. Adhesive wear is as ambiguously defined as is sliding wear, though the two are not strictly synonymous. Adhesive wear denotes a wearing action in which no specific agency can be identified as the cause of the wear.

Prevention of Adhesive Wear:-

The following guidelines are recommendations to prevent adhesive wear in metals, polymers, and ceramics:

- Avoid sliding similar materials together, particularly metals
- If fatigue due to repeated high-contact pressure is not likely to be a problem, then high hardness is a desired property. However, avoid sliding hard metals against hard metals in lubricated systems to avoid scuffing and to accommodate debris
- Consider the effect of relative hardness of phases in materials. For example, a high-chromium cast iron may have a hardness of 400 HB, which is moderate. However, that cast iron may contain Cr7C3, which has a hardness of about four times that of 400 HB and will damage the counter surface considerably. The same applies to polymers, which seem rather soft relative to metals. However, wear-resisting polymers often contain glass or some other hard filler that wears metal counter faces rather severely. Hard phases in one body may fragment and become embedded in the counter face, which causes abrasion if the fragments extend above the surface.
- Even if done inadequately, lubrication will reduce wear. Some lubrication can be applied by providing an atmosphere that is corrosive in order to form surface films, many of which produce lower friction than if that film were not present.[10]

1.8.3.3 CORROSIVE WEAR

It is defined as the degradation of materials in which both corrosion and wear mechanisms are involved. The combined effects of wear and corrosion can result in total material losses that are much greater than the additive effects of each process taken alone, which indicates synergism between the two processes. This article focuses on the corrosion-wear synergism in aqueous slurry and grinding environments. Examples of corrosive wear problems encountered in industry are provided, and the effects of environmental factors on corrosive wear are discussed. [10]

1.8.3.4 OXIDATIONAL WEAR

It is a form of wear that primarily occurs during nominally unlubricated conditions of sliding. It is usually, but not always, a beneficial form of corrosion in which oxide films that are approximately 3 or 4 m (120 or 140 in.) thick form on the real areas of contact at the relatively high "hot-spot" temperatures that often occur between sliding interfaces. In many cases, tribological oxidation can reduce the wear rate by two orders of magnitude, compared with the wear of the same metals under an inert atmosphere. However, amelioration of the wear rate will only occur if the oxide layers are formed during sliding. It is not possible to artificially produce low-wear surfaces by preoxidizing the surfaces under static furnace conditions. Oxidational wear can also occur under lubricated sliding conditions when the oil film thickness is less than the combined surface roughnesses of the triboelements, for example, under conditions of boundary lubrication. Oxidational wear is the "last defense" that a lubricated metal surface has against scuffing. It is also the only defense that some of the recently emerging ceramic tribosystems have against failure when running at high temperatures. [10]

CHAPTER –II

2. LITERATURE SURVEY:

2.1 PREPARATION OF PARTICULATE COMPOSITES:

The particulate composition can be prepared by incorporating the reinforcing particles directly into the matrix or by producing in-situ with the help of various techniques. The first approach is more popular, where the reinforcing particles are injected into either solid or liquid matrix.

In case of powder metallurgy technique, alloy powders are blended with the ceramic particles and the mixture are hot or cold compacted in controlled atmosphere to desire shape and degassed. The compact are usually hot worked for final consolidation. In case of cold compact, sintering may precede the hot working operation. But the liquid phase processes have been investigated in greater detail in recent year due to its ability to produce massive component at lower cost. [11] In these processes, the ceramics particles are incorporating into metal using various techniques or cast into ingots for secondary processing. The major difficulties in such processes are the non wettability of the particle by liquid metal and the consequent rejection of the particle from the melt and also the non uniform distribution of particles due to preferential segregation and extensive interfacial reaction.

It has been reported by various worker [12, 13, 14, 15] that that during solidification of composite, particle-interface interaction plays a major role in dictating the particle distribution. The particle ahead of the interface may get pushed, engulfed, or entrapped in the moving solidification front. In turn the presence of the particle may influence the morphology of the advancing interface. Dutta et al, [16] Rohatgi et al [17] and Stefanescu [18] et al. have observed that the presence of silicon carbide particle can significance alter the morphology of the advancing interface during directional solidification of Al-alloy matrix composites. Dutta and Surappa [19] presented and analysis of the modified solute concentration field and temp. Subsequently the effect of these two fields on the morphology of the dendrites has also been estimated. These changes in the morphology will have an important bearing on the particle distribution in the solidified structure, which has also been discussed by them.

In the early stage of development of cast particle composite, particle were injected into molten aluminium through carrier gas.[20] To achieve greater recovery, coating of particle has been suggested than the uncoated particle e.g. nickel coating of graphite particle in case of Al matrix composite . Nickel improved the wettability of the particle by Al melt. To ensure uniform distribution of particles, stirring of melt after injection has been suggested.[20]

Pellet method was developed to incorporate ceramic particle in to the Al melt. [21, 22] In this method the coarser particle of the base alloy and reinforcing ceramic particle were mixed and pressed to form pellets. These pellets were subsequently plunged into the melt followed by the slow stirring manually or mechanical. The distribution of particles in the cast particulate composites was not satisfactorily uniform when it was prepared by injection technique or pellet method without stirring. To overcome such difficulty." Vortex method" was developed. [23, 24] this is the most common method used at present for "cast metal matrix particulate composite ". in this method, molten metal in a crucible is stirred with a suitable impeller to form vortex in the melt. Required amount of ceramic particles are slowly added into the vortex at a predetermined rate. The particles get entrapped within the melt and distribute uniformly in it during stirring. The major difficulty in such a method is the lack of wettability of the particles by the liquid AL and the rejection of particle from the melt.

Addition of Mg, Li, Si, and Ca into Al melt improved wettability [25] either by changing the interfacial energy through some interfacial reaction or by modifying the oxide layer on the metal surface. Detailed investigation has been carried out by Pai et al [26] to study the effect of Mg addition on the recovery of alumina particles in Al melt. It is also revealed in their study [26], that fresh addition of Mg is more effective as compared to pre alloyed Mg. in recent study it has been demonstrated that addition of Ca also improve the wettability of alumina particle in Al-4.5 Cu melt, but its effect on improvement of the retention of particle is less then with magnesium addition. [26]

Another approach to avoid the rejection of ceramic particle from AL melt is to add the particle into partially solidified slurry of liquid alloy.[27] On the basis of phase diagram of the alloy. A suitable temperature is selected to have about 40% of solid in the alloy. The partially solid alloy is agitated and the particles are added to it. Initially the ceramic particles are mechanically entrapped in the partially solidified slurry and with further mixing, interaction

between the ceramic particle and the liquid alloy promote binding. After mixing of the ceramic particle, the semisolid composite slurry can be cast directly by rheocasting. In another method, the composition slurry is heated to temperature above its liquid temperature and subsequently can be cast. This process is known as compocasting. [28] A similar method has been used to prepare Al –alumina particle composites [29] by addition the particle into a mechanically stirred Al alloy in the semi fused state. The process requires costly tooling and still, uniform distribution of particle is difficult to obtain. In case of vortex method, heat treatment of particle prior to addition into the molten alloy has been suggested to improve the retention of the particles in the melt.[30]Uncoated graphite particles could be introduce into Al melt at a temperature above the liquidus (UPAL process) the prior heat treatment of the particle removed absorbed gases and the volatile substance from the surface of the particle, and the hence increase the wettability of the particle. In case of alumina particle, prior heat treatment of the particle at around 900 ˚C dehydroxylated the surface of the particle and hence increase the surface energy.[23] Probably this increase surface energy improved the wettability of the particle in liquid Al.

Osprey process has been developed to prepare Al matrix particle composite [30]. In this process, the reinforcement particle is introduced into a stream of molten alloy, which is subsequently atomized with a jet of inert gas. The sprayed mixture is solidified on a substance in the form of pellet. In a recent study, stray formed Al-SiC composite have been thixoformed [31] to avoid segregation of the particle and detrimental interfacial reaction associated with liquid phase process. Variable co-deposition of multiphase material (VCM) is a technique [30,32]similar to osprey process . it ensure better control on the interfacial reaction between reinforcing particle and the matrix and promotes a rapidly solidified fine grained matrix. In this process, reinforcing particles are injected at a specific level of the spray atomized metal containing limited volume traction of solid. So that the reaction between the matrix and the reinforcement particle are negligible. The particle can also be introduced into the liquid alloy to spray deposition. [30]

In summary it can be conclude that although many methods have been tried to prepare particle melt slurry, vortex method appears to be the most convenient and amenable to a large-scale production of the slurry. The slurry produced can be cast different shape or starting material for secondary production.

CHAPTER-III

3.0 EXPERIMENTAL WORK:

3.1 STIR CASTING:

The primary objective of the present work is to form reinforcing particles within the Al melt by addition of iron oxides into the base metal in the liquid state. In the first part of the work attempt is made to prepare the composites and to characterize them by identifying the various compounds that has been formed with in the matrix In the second part, the physical and mechanical properties of the composites are reported.

3.1.1 CHOICE OF MATERIALS:

3.1.1.1 CHOICE OF MATRIX:

Commercially pure Al was taken on the matrix at the first stage of experiments so that oxidation of Al matrix could produce fine particles of Al2O3 within the matrix. Initial results with pure Al matrix showed that desired uniformity of the particle distribution could not be obtained with commercially pure Al melt The reaction was also minimal There was large amount of particles left out in the Crucible itself .For improving wettability and this to facilitate reaction and also to obtain a uniform distribution of reinforcing particles, addition of Mg to the melt was tried.

3.1.1.2 CHOICE OF OXIDE PARTICLES:

To form Al2O3 or MgAl2O4 in-situ particles in Al matrix by the oxidation of pure Al or Al-2%Mg alloy respectively iron oxide was selected for addition into the melt. The iron oxide lie above the line of Al2O3 and MgO in the Ellingham diagram, so the iron oxide when added to Al, would react with the melt and form Al2O3, MgO etc.

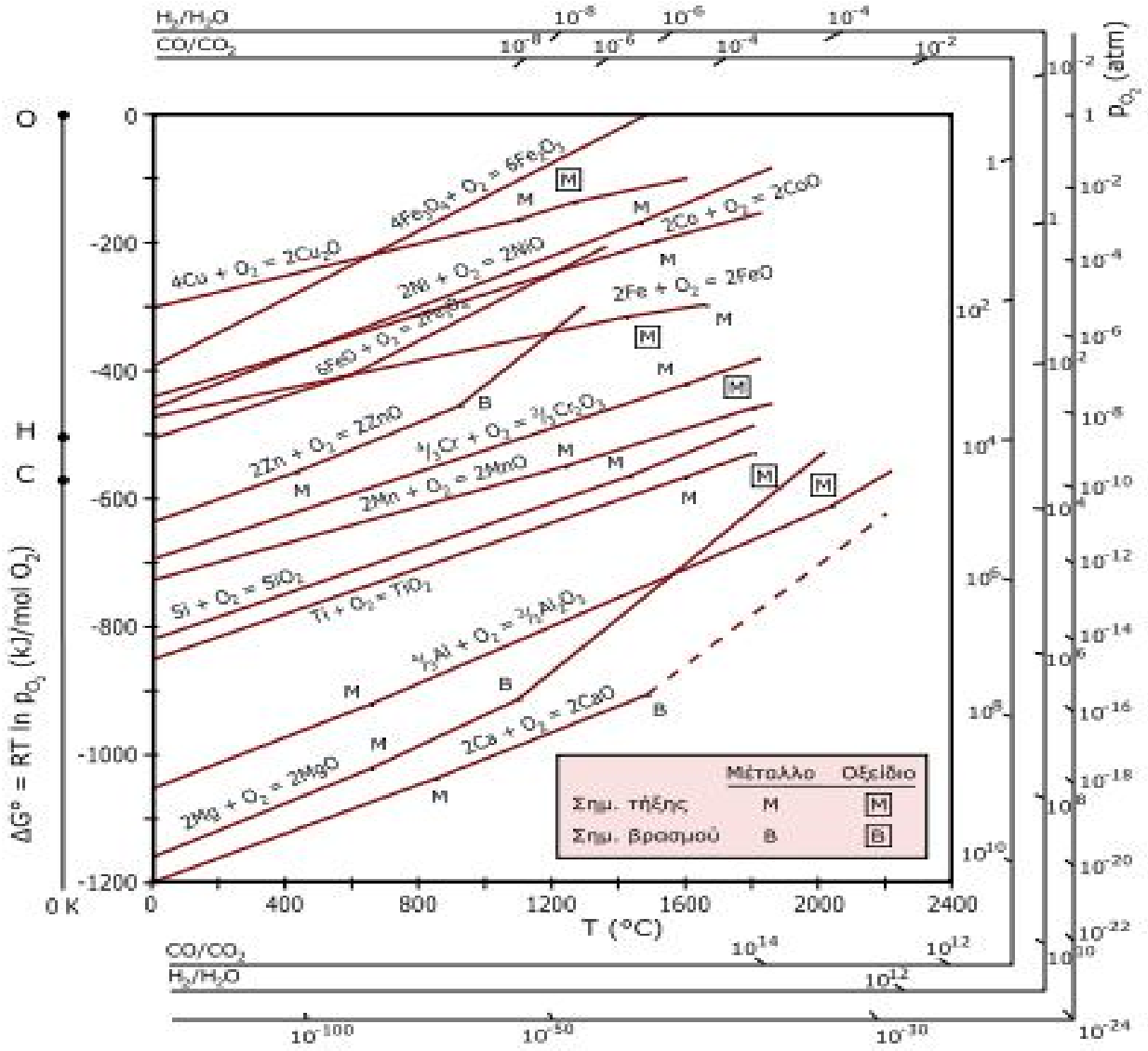


Fig 3.1 Temperature versus standard free energy of formation of oxides

3.1.2 MELTING AND CASTING

The aluminum- iron ore metal matrix composite was prepared by stir casting route. For this we took 500gm of commercially pure aluminum and desired amount of iron ore in powder form. The Fe ore particle was preheated to 300^0C for three hours to remove moisture. Commercially pure aluminum was melted in a resistance furnace. The melt temperature was raised up to 720^0C. Then the melt was stirred with the help of a mild steel turbine stirrer.

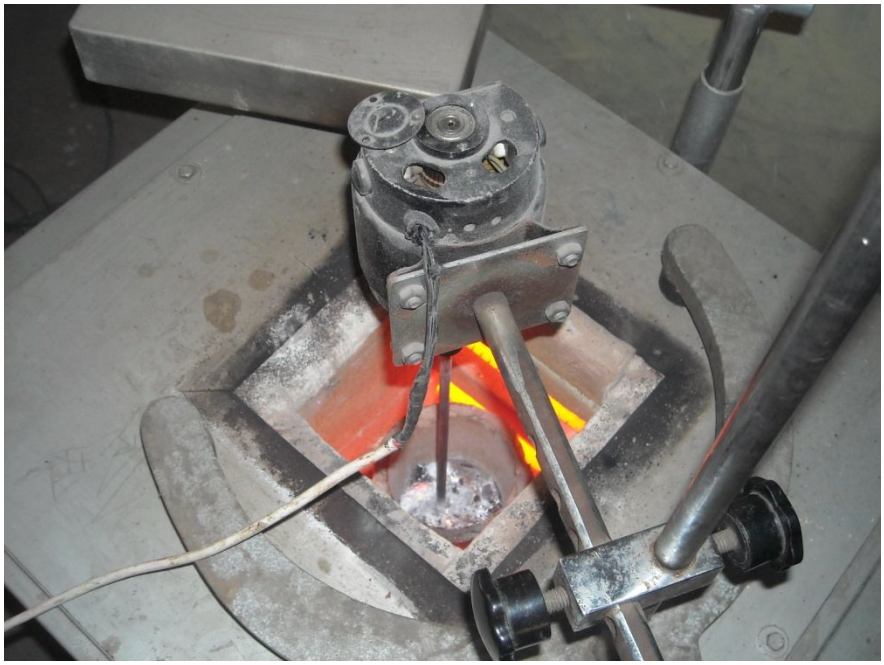


Fig.3.2 Laboratory stir casting set up

The stirring was maintained between 5 to 7 min at an impeller speed of 200 rpm .The melt temperature was maintained 700^0C during addition of Mg and Fe ore particles. The dispersion of Fe ore particles were achieved by the vortex method. The melt with reinforced particulates were poured into the preheated permanent metallic mold. The pouring temperature was maintained at 680^0C.The melt was then allowed to solidify in the mould.

3.2 MICROSTRUCTURAL CHARACTERIZATION:

3.2.1 OPTICAL MICROSCOPY

The casting procedure was examined under the optical microscope to determine the cast structure .A section was cut from the castings. It is first belt grinded followed by polishing with different grade of emery papers. Then they were washed and polished in clothes and then washed, dried and etched with Keller's solution and then examined though optical microscope.

3.2.2 SCANNING ELECTRON MICROSCOPY

Microstructural characterization studies were conducted to examine distribution of reinforcement throughout the matrix. This is accomplished by using scanning electron microscope. The composite samples were metallographically polished prior to examination. Characterization is done in etched conditions. Etching was accomplished using Keller's reagent. The SEM micrographs of composites were obtained using the scanning electron microscope. The images were taken in secondary electron (SE) mode. This analysis was done by a JEOL 6480 LV scanning electron microscope (SEM) equipped with an energy dispersive X-ray (EDX) detector of Oxford data reference system.

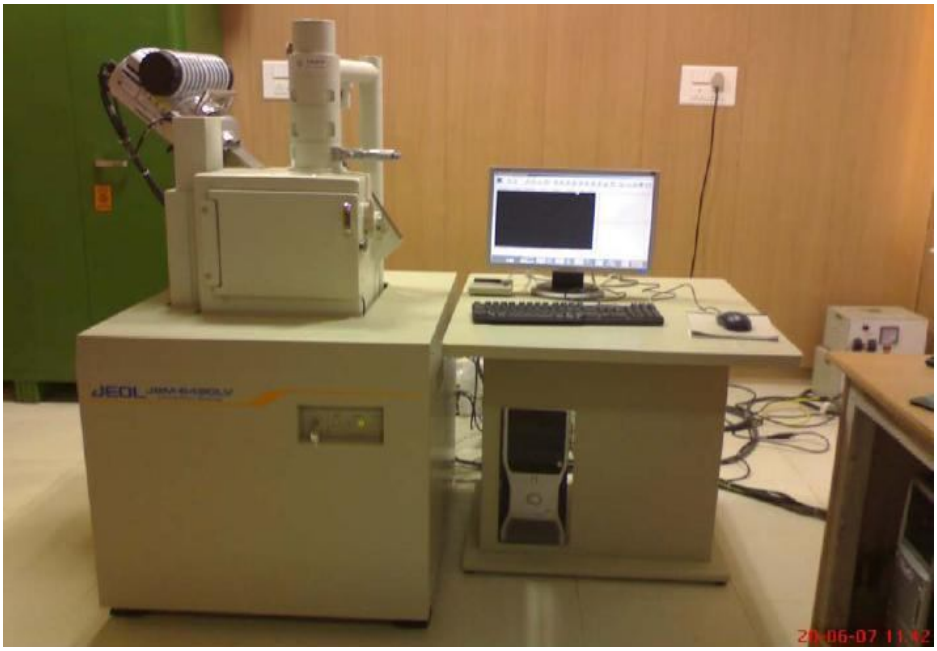


Fig.3.3 JEOL JSM-6480LV scanning electron microscope

3.3 PARTICLE SIZE ANALYSIS

Particle size of the iron ore powder was measured by Malvern particle size analyzer (Model Micro-P, range 0.05-550 micron). Firstly, the liquid dispersant containing 500 ml of distilled water was kept in the sample holder. Then the instrument was run keeping ultrasonic displacement at 10.00 micron and pump speed 1800 rpm.

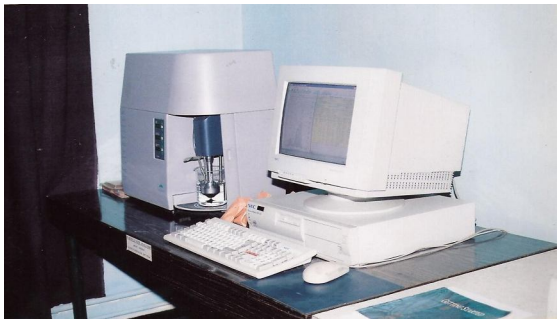


Figure 3.4: Malvern particle size analyzer

3.4 MECHANICAL PROPERTIES OBSERVATION

3.4.1 HARDNESS

Hardness measurements were carried by using standard Vickers hardness test machine. The indenter used for this measurement was diamond indenter and the load applied was 5kg for 10 minute.

3.4.2 WEAR BEHAVIOUR

A Ducom, Bangalore made computerized pin on- disc wear test machine was used for these tests. The wear testing was carried out at a different sliding velocity with different normal loads. A cylindrical pin of size 1.1cm diameter and 2.1cm length, prepared from composite casting, was loaded through a vertical specimen holder against horizontal rotating disc. The rotating disc was made of carbon steel of diameter 50 mm and hardness of 64 HRC. The weights were measured before and after each test segment to determine the abrasive wear loss of each sample. Scanning electron microscopy was used to analyze the morphology of the worn surfaces of sample.

CHAPTER-IV

4.0 RESULTS & DISCUSSION

4.1 PARTICLE SIZE ANALYSIS OF IRON ORE

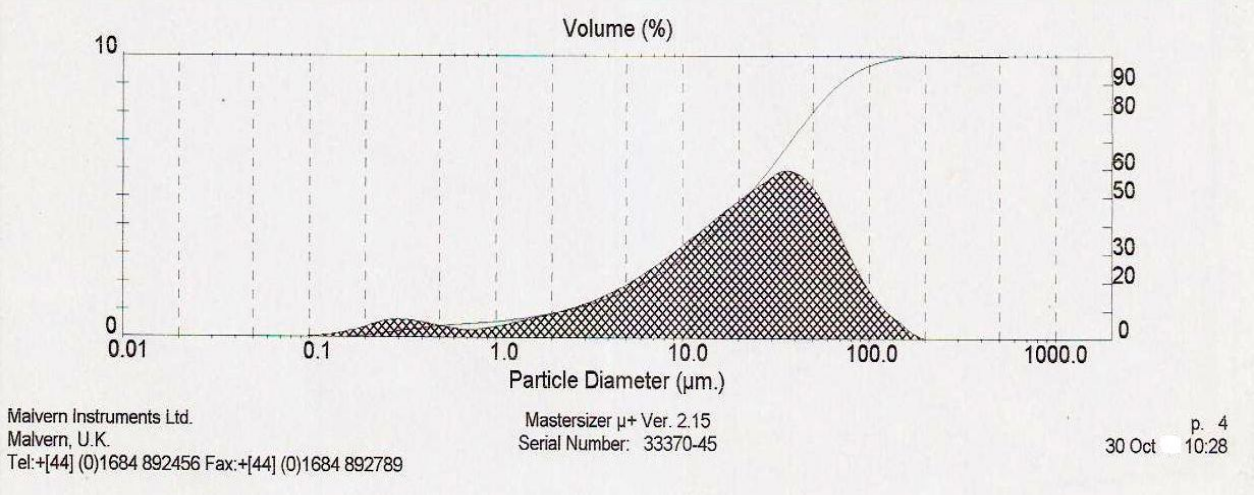


FIG 4.1 PARTICLE SIZE ANALYSIS

As seen from the particle size analysis there are very fine particles as well as coarse ones (1-100 µm). Thus the strengthening of composite can be due to dispersion strengthening as well as due to particle reinforcement.

4.2 HARDNESS MEASUREMENT

Composite samples	Hardness
Pure aluminium	
Al-2%Mg-10% Fe ore	
Al-5% Fe ore	

The above table shows that incorporation of iron ore particles in Aluminium matrix causes reasonable increase in hardness. The strengthening of the composite can be due to dispersion strengthening as well as due to particle reinforcement.

4.3 WEAR BEHAVIOUR:

Wear behavior of different composites were studied with different parameter like sliding distance, sliding velocity, and applied load.

4.3.1 EFFECT OF SLIDING DISTANCE

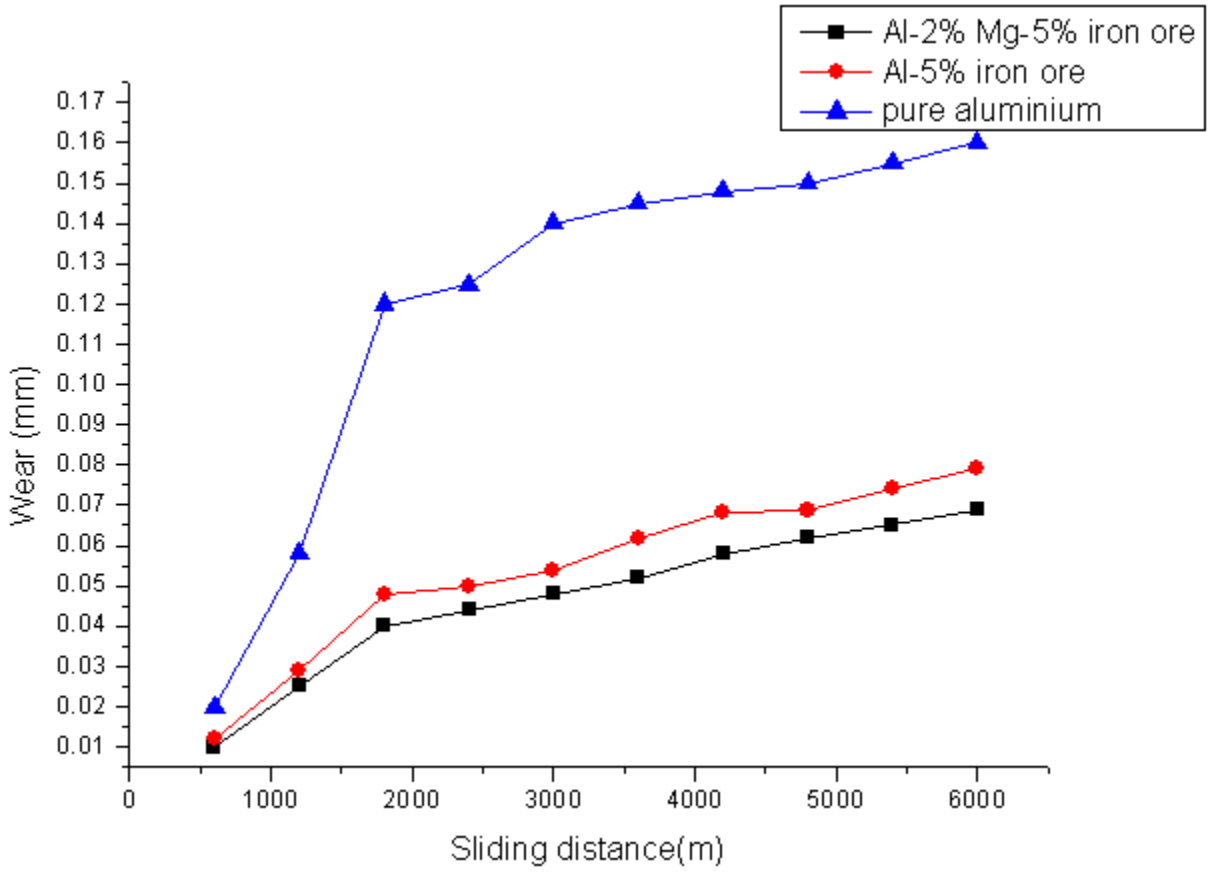


Fig 4.2 Effect of sliding distance on weight loss of composites

The above figure shows the relationship between weight loss and sliding distance. For all the compositions the wear rate is high initially which is followed by a steady wear rate. Bulk wear rate depends on amount of dispersed phase present in the composite. Addition of magnesium decreases the wear rate significantly in comparison to as cast aluminium. The wear rate has increased with sliding distance. The reason being the oxide particles are removed giving rise to

three body wear and wear rate increases with increase in sliding velocity. The reason being the oxide particles are removed giving rise to three body wear.

4.3.2 EFFECT OF APPLIED LOAD

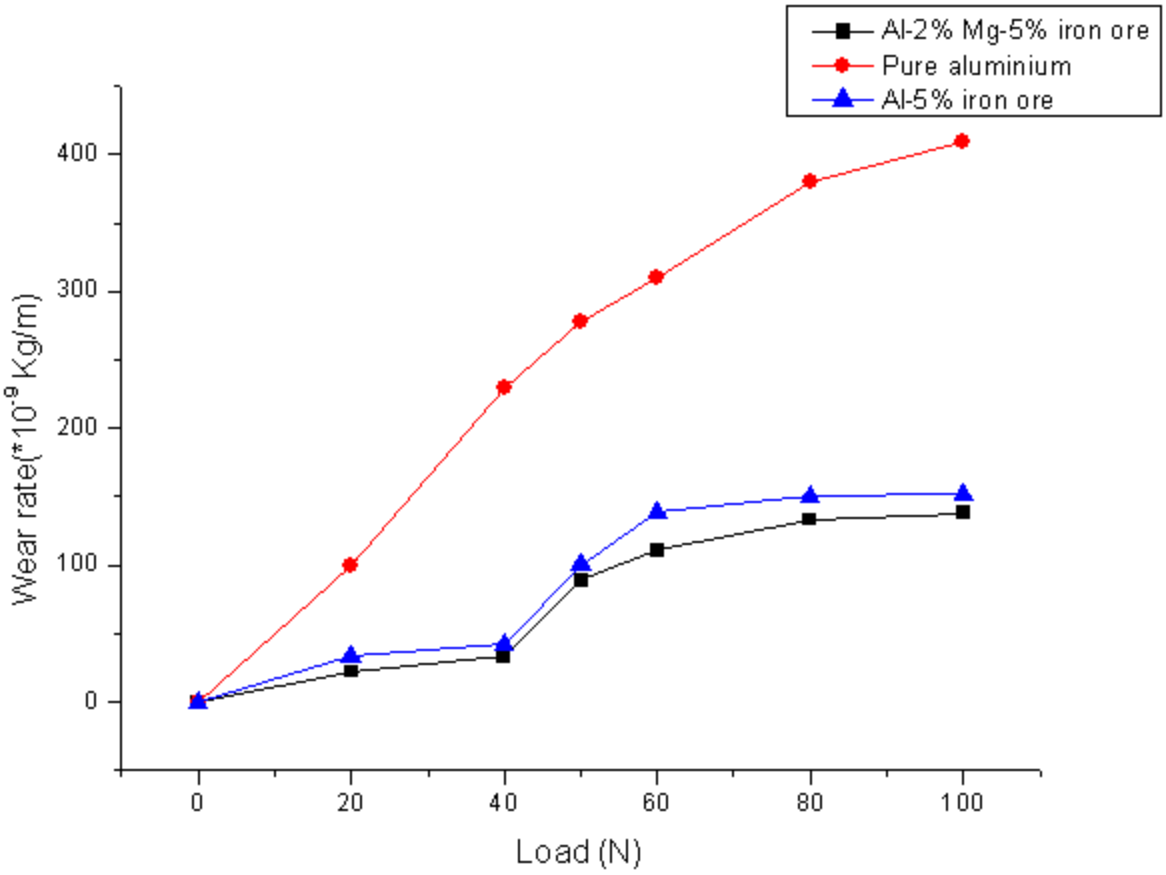


Fig 4.3 Effect of varying load on wear rate of different composites at sliding velocity of 1m/s.

Above figure shows the relation between applied load and wear rate of the composites at a constant sliding velocity of 1 m/s. It is seen that at lower load the wear rate is low in case of composite then it increases slowly. The rate of wear in case of as cast aluminium sample is extremely high in comparison to composite.

4.3.3 EFFECT OF SLIDING VELOCITY ON WEAR RATE

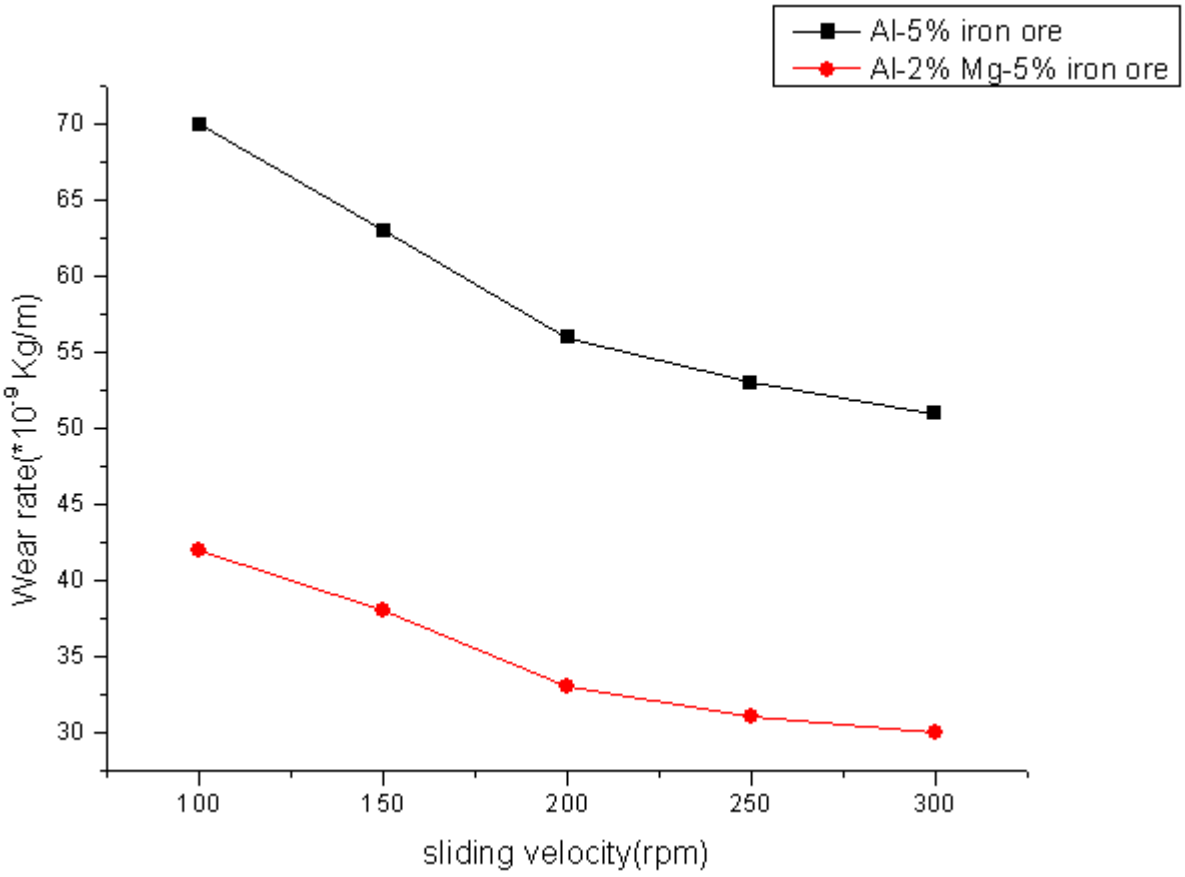


Fig 4.4 Effect of sliding velocity on weight loss on at 30 N load

Above figure shows the relation between sliding speed and wear rate of the composites at a constant load of 30 N. The above figure clearly shows that change of wear rate initially high and after a certain sliding velocity the change of wear rate becomes constant. . Like variation of wear rate with load in the in the present case also increase in dispersed phase reduces the wear rate.

4.4 SEM MICROGRAPHS OF THE MMCs

SEM micrographs were taken to analyze the surface of Al-2% Mg-10% Iron ore composites.

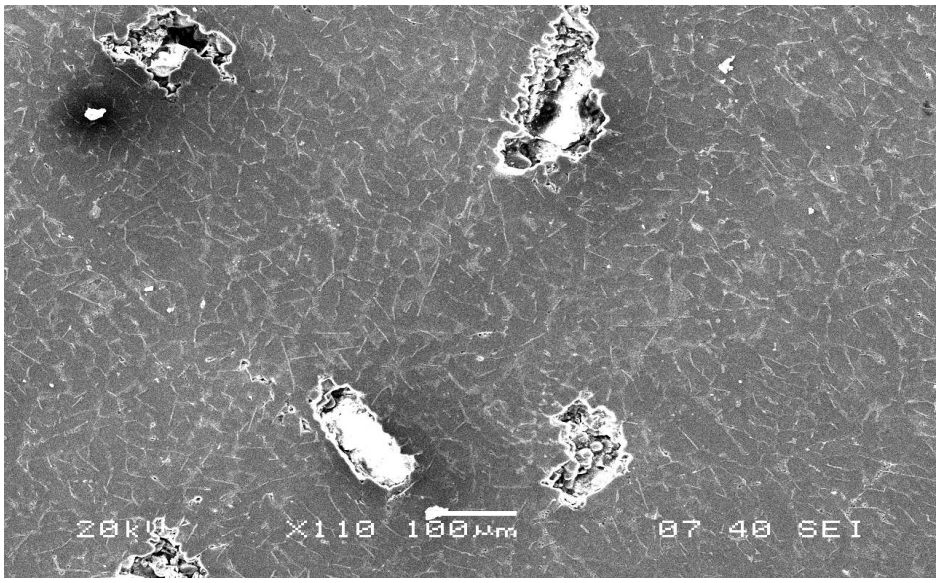


FIG 4.5 (a) SEM micrograph of Al-2% Mg-10% iron ore composite

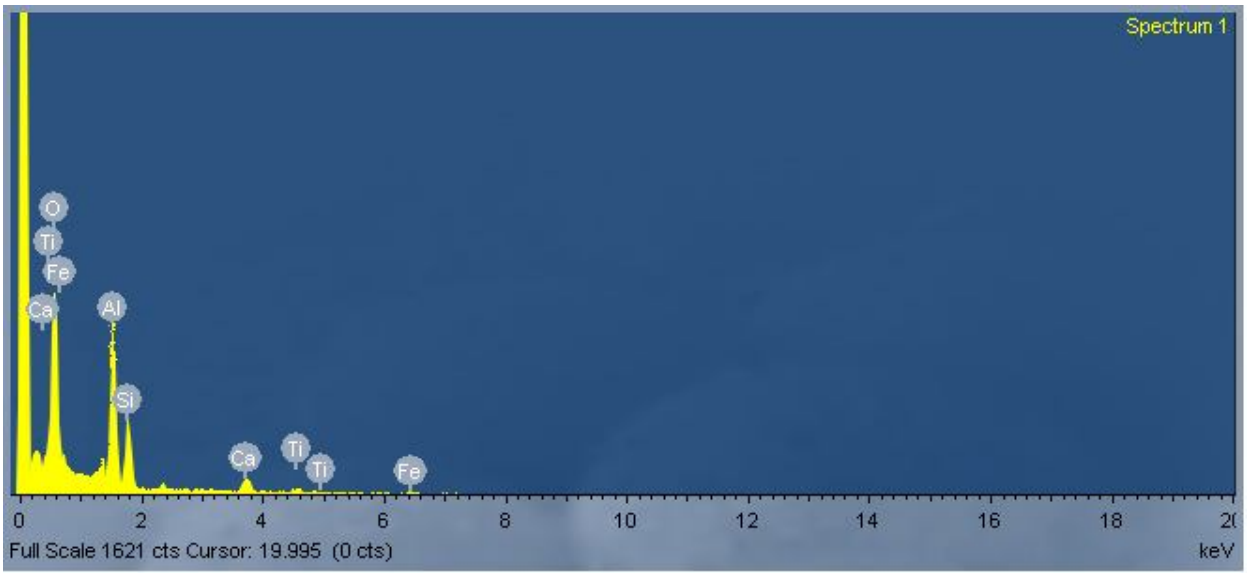


FIG 4.5 (b) EDS microanalysis for Al-2% Mg-10% iron ore composite

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
O K	55.02	1.0462	53.72	1.76	66.35
Mg K	12.31	0.8067	15.60	0.86	12.67
Al K	11.51	0.7068	16.64	0.90	12.19
Si K	6.27	0.6603	9.70	0.68	6.82
Ca K	2.42	0.9595	2.58	0.36	1.27
Ti K	1.03	0.8088	1.31	0.35	0.54
Fe K	0.37	0.8230	0.46	0.41	0.16
Totals			100.00		

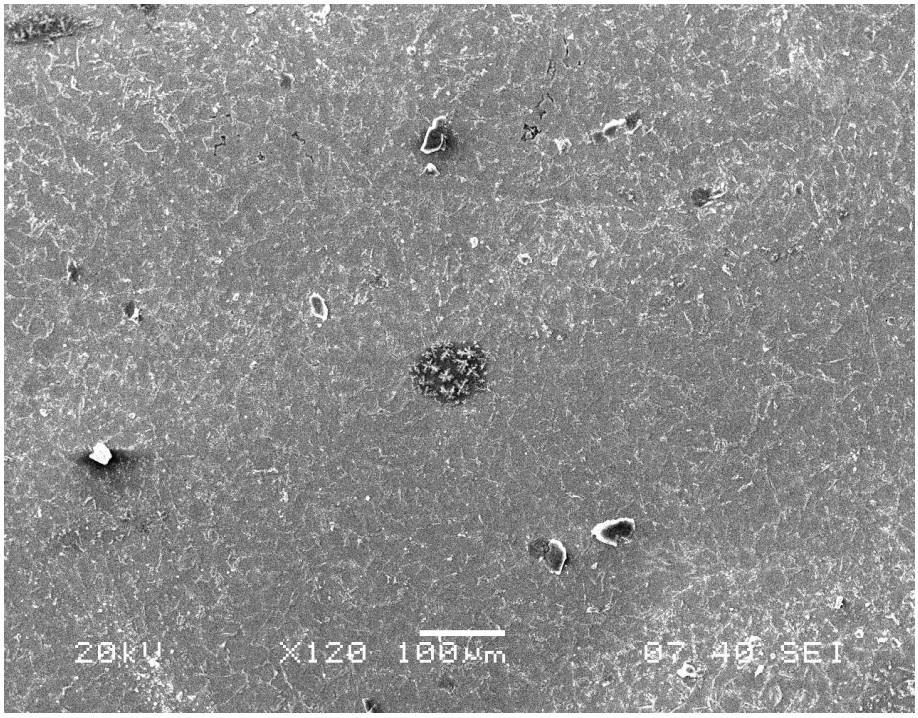


FIG 4.6 (a) SEM micrograph of Al-2% Mg-10% iron ore composite

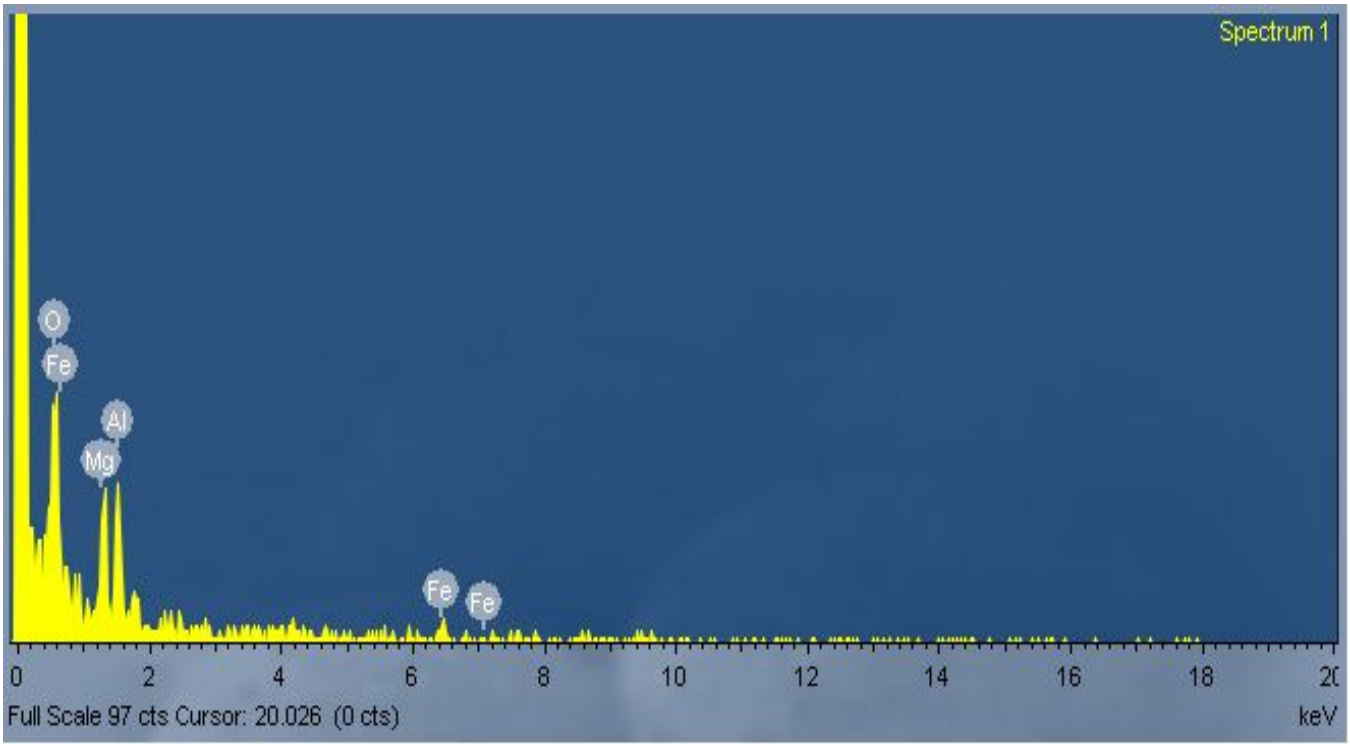


FIG 4.6 (b) EDS microanalysis for Al-2% Mg-10% iron ore composite

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
O K	32.96	1.3715	49.54	9.08	63.70
Mg K	7.18	0.7442	19.87	5.20	16.82
Al K	6.40	0.6321	20.86	5.28	15.90
Fe K	3.96	0.8401	9.72	6.68	3.58
Totals			100.00		

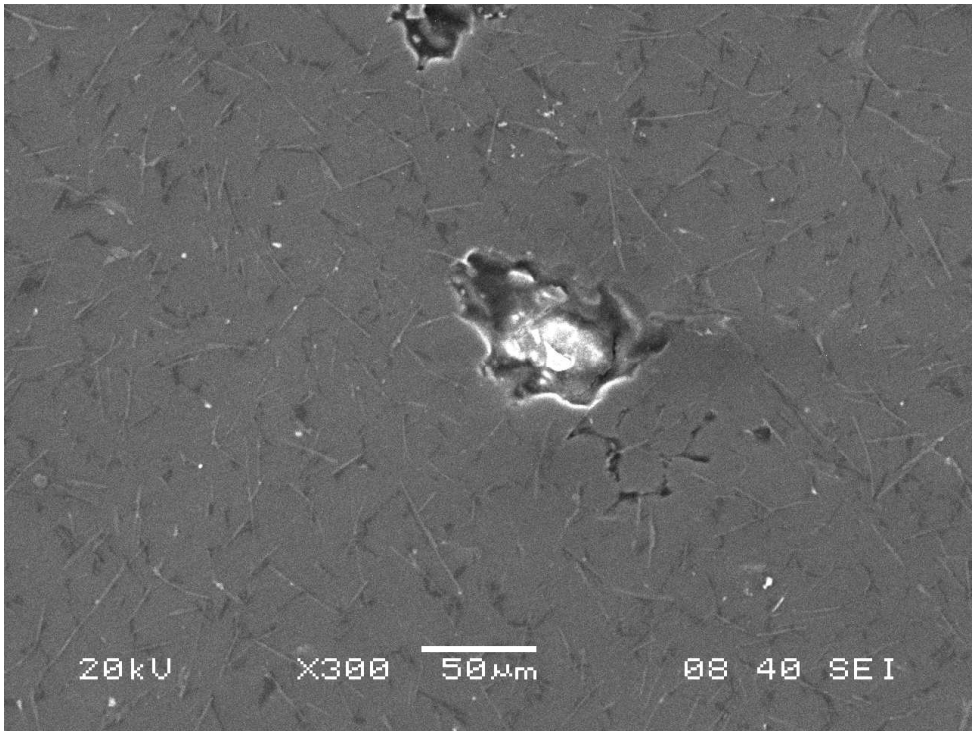


Fig 4.7 (a)SEM micrograph of Al-2% Mg-10% iron ore composite

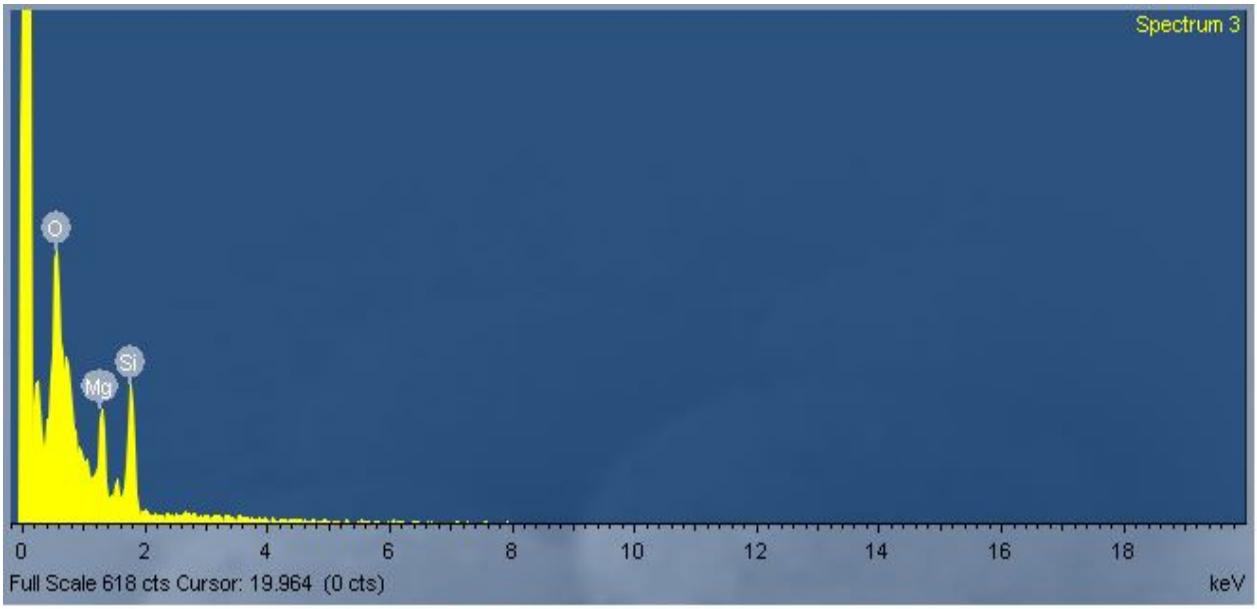


Fig 4.7(b)EDS microanalysis for Al-2% Mg-10% iron ore composite

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
O K	25.63	1.2049	58.44	4.31	69.94
Mg K	4.75	0.7948	16.40	2.35	12.91
Si K	7.30	0.7951	25.16	2.87	17.15
Totals			100.00		

Thermodynamic analysis indicates that there is a possibility of chemical reaction between aluminium melt, iron ore, magnesium and silica present in commercially aluminium. The chemical reduction occurs, as follows:

$3Mg\ (l) + Fe_2O_3 = 3MgO + 2Fe$

$2Mg\ (l) + SiO_2 = 2MgO + Si$

$Al\ (l) + Mg\ (l) + 2O_2 = MgAl_2O_4$

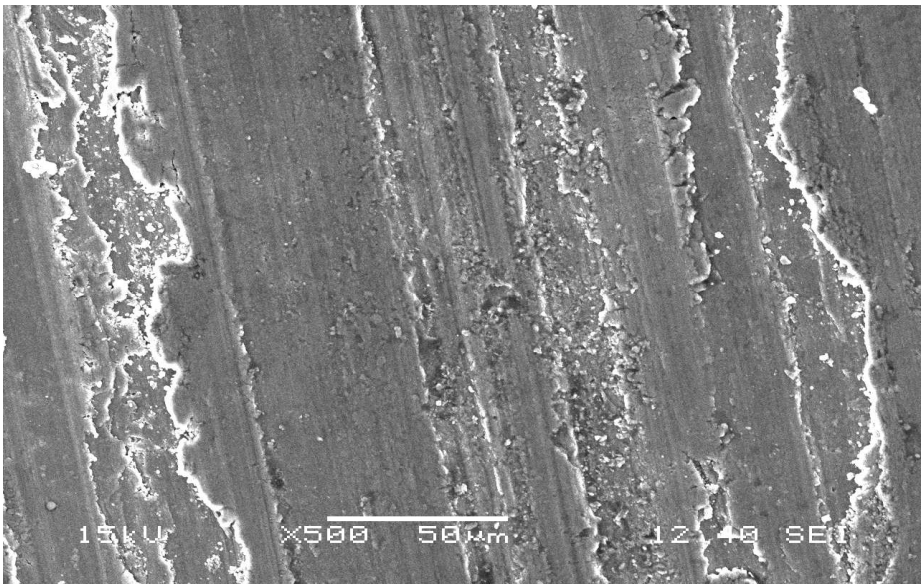
$2Al\ (l) + Fe_2O_3(s) = 2Fe(s) + Al_2O_3(s)$

$2Al\ (l) + 3/2\ SiO_2(s) = 3/2\ Si(s) + Al_2O_3(s)$

The elements (Si and Fe) formed by reduction reaction would alloy with the matrix. Gibbs free energy and the heats of reactions are highly exothermic in nature. As a result of the above reaction greater amount of eutectic silicon is seen in the composites in the shape of needle throughout the matrix.

SEM micrographs of Al-2% Mg-10% Fe ore are shown in figure 4.5(a), 4.6(a) and 4.7(a).The spot analysis at different location indicate the presence of Al_2O_3 and MgO.

4.5 SEM MICROGRAPHS OF THE WORN SURFACE



(b)

Fig.4.8 SEM micrographs of the worn surface of Al-5% iron ore

SEM micrograph of the worn surface of Al-5% iron ore in-situ composite is shown in the above figure. Most of the grooves are parallel to the sliding direction; it is evident from the worn pins. Such features are characteristics of abrasion, in which hard asperities of the steel counter face, or hard reinforced particles in between the contacting surfaces, plough or cut into the pin, causing wear by the removal of small fragments of material.

CHAPTER-V

5.1 CONCLUSIONS:

The following conclusion may be drawn from the present work:

1. From the study it is concluded that we can use low grade iron ore for the production of in-situ composites.

2. Alumina, magnesia particles can be produced in-situ by reaction between iron oxides with Al and Mg.

3. Addition of Mg improves the wettability of iron with Al melt and thus increases the retention of low grade iron ore to some extent in the matrix.

4. The wear resistance and hardness has improved significantly with addition of low grade iron ore.

5. Increased normal load and sliding velocity increases magnitude of wear.

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